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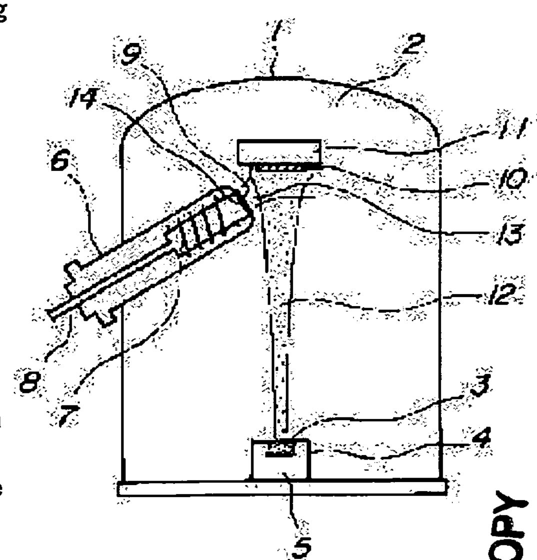
(54) FORMATION OF OXIDE OPTICAL THIN FILM AND FORMING DEVICE OF OXIDE OPTICAL THIN FILM

(57)Abstract:
PROBLEM TO BE SOLVED: To provide a device capable of forming an oxide optical thin film having excellent optical characteristics without forcedly heating the object to be film-formed such as a substrate.
SOLUTION: In a forming device of an oxide optical thin film provided with a 1st vacuum tank generating oxygen plasma by high-frequency discharge and a 2nd vacuum tank connected with the 1st vacuum tank and which inside under a vacuum higher than the case of the 1st vacuum tank is set with the object to be film-formed and an evaporating source 3 evaporating evaporating particles 12 onto the object 10 to be film-formed by electron beams, the 1st vacuum tank is set with a dielectric board 1 provided with an orifice 14 of ≤0.3 mm diameter, the oxygen plasma 13 is applied on the object to be film- formed as a plasma flow dominant over the oxygen radical by interposing the orifice, and the evaporating particles are passed through the plasma flow, and they are mixed to execute film formation.



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CLAIMS

[Claim(s)]
[Claim 1] While the 1st vacuum tub which sets it as a pressure predetermined by passing oxygen, and generates the oxygen plasma by high frequency discharge, and this 1st vacuum tub are connected In the formation equipment of the oxide optical thin film equipped with the 2nd vacuum tub by which the object formed membranes and the evaporation source which evaporates an evaporation particle to said lifter formed membranes with an electron beam were installed in the interior set as the high vacuum from the 1st vacuum tub The dielectric plate which prepared the orifice of 0.3mm or less of diameters in said 1st vacuum tub is installed. The lifter in said 2nd vacuum tub formed membranes is irradiated as a plasma style dominant in an oxygen radical by minding said orifice for the oxygen plasma generated by high frequency discharge in said 1st vacuum tub. Formation equipment of the oxide optical thin film characterized by forming membranes by passing and mixing this plasma style for said evaporation particle.
[Claim 2] Formation equipment of the oxide optical thin film according to claim 1 characterized by forming said 1st vacuum tub and the dielectric plate with at least one ceramic ingredient chosen from boron nitride, boro-silicated glass, a quartz, oxidation aluminum, and nitriding aluminum.
[Claim 3] Claim 1 characterized by the oxygen radical concentration irradiated by the lifter in said 2nd vacuum tub formed membranes being more than 1016-piece [/cm] 2 and s, or formation equipment of an oxide optical thin film according to claim 2.
[Claim 4] The formation approach of the oxide optical thin film characterized by making into an evaporation source the evaporation material which becomes either of claim 1 to claims 3 from a metallic oxide using the formation equipment of the oxide optical thin film of a publication, and forming membranes to the lifter which does not carry out forcible heating formed membranes.

[Translation done.]

* NOTICES *

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2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the formation approach of an oxide optical thin film of having high performance, and the formation equipment of an oxide optical thin film.
[0002]
[Description of the Prior Art] Ta 2O5, Nb 2O5, In 2O3, SiO2, and TiO2 etc. -- the oxide thin film is used for a multilayer antireflection film, the reflective increment film, the interference filter film, etc. as an optical thin film. Such [conventionally] an oxide thin film was produced by reaction vacuum deposition. This approach is an approach of producing oxygen gas and oxidation reaction and making the evaporation particle evaporated with resistance heating or an electron beam from the vacuum evaporationo ingredient forming on an elevated-temperature substrate 300 degrees C or more.
[0003] In recent years, in order to produce the oxide optical thin film of high performance more, an evaporation particle and oxygen gas were made to react all over a plasma field, and the plasma assistant vacuum deposition which forms membranes was proposed. This approach uses the active state of the plasma and measures acceleration of oxidation reaction at low substrate temperature, and improvement in adhesion with a substrate more.
[0004] For example, the arc discharge plasma assistant vacuum deposition (JP,9-71857,A etc.) which the seal of approval of the high-frequency voltage is carried out, the plasma is generated by glow discharge, arc discharge is generated from a plasma gun including RF plasma assistant vacuum deposition which raises the adhesion and the precision to a substrate of a thin film, or the cathode which consists of LaB6 which is easy to generate a thermoelectron recently (6-ized boron lanthanum), the plasma style of high density is formed, and a vacuum evaporationo ingredient is evaporated, and is made to form is proposed.
[0005]
[Problem(s) to be Solved by the Invention] However, the following troubles are mentioned in the plasma assistant vacuum deposition shown above.
(1) Many properties of an optical thin film deteriorate by the film surface impact of high energy particles, such as ion in the plasma, (attenuation of the transparency by the increase of surface roughness etc.).
(2) Compared with the usual vacuum deposition method, the film consistency of the thin film with which it is hard to obtain a high degree of vacuum, and it is formed becomes low.
(3) In order to raise oxidation reactivity and a film consistency, the elevated temperature 300 degrees C or more was impressed to the substrate.
For this reason, the object for membrane formation (object formed membranes) was restricted to the ingredient with thermal resistance.
[0006] The 1st object of this invention is offering the equipment which can form the oxide optical thin film which has the outstanding optical property, without carrying out forcible heating of the object formed membranes, and the 2nd object of this invention is offering the approach of forming easily the oxide optical thin film which has the outstanding optical property, without carrying out forcible heating of the object formed membranes.
[0007]
[Means for Solving the Problem] Invention of claim 1 for solving said technical problem While the 1st vacuum tub which sets it as a pressure predetermined by passing oxygen, and generates the oxygen plasma by high

frequency discharge, and this 1st vacuum tub are connected In the formation equipment of the oxide optical thin film equipped with the 2nd vacuum tub by which the object formed membranes and the evaporation source which evaporates an evaporation particle to said lifter formed membranes with an electron beam were installed in the interior set as the high vacuum from the 1st vacuum tub The dielectric plate which prepared the orifice of 0.3mm or less of diameters in said 1st vacuum tub is installed. The lifter in said 2nd vacuum tub formed membranes is irradiated as a plasma style dominant in an oxygen radical by minding said orifice for the oxygen plasma generated by high frequency discharge in said 1st vacuum tub. It is formation equipment of the oxide optical thin film characterized by forming membranes by passing and mixing this plasma style for said evaporation particle.
[0008] The example used for membrane formation by using a radical as an oxidation labile kind is accepted well in recent years. In a typical example, there is a radical beam method represented by JP,8-60347,A. However, as for this method, space has linked even the substrate (object formed membranes) directly from the source of the RF plasma. Therefore, since the active species which may relate to membrane formation, such as not only a radical but ion, ultraviolet rays, etc., is irradiated by the substrate (object formed membranes), the alternative exposure of a radical is barred.
[0009] In the formation equipment of the oxide optical thin film of this invention, since the dielectric plate which prepared the orifice of 0.3mm or less of diameters in from the source of the RF plasma before the object formed membranes is installed and the radical is irradiated through this orifice, a radical can be more nearly selectively irradiated to the object formed membranes.
[0010] Moreover, in the formation equipment of the oxide optical thin film of this invention, it is that the inside of the tub which intervenes and forms the dielectric plate with which the 1st vacuum tub and the 2nd vacuum tub have said orifice is maintained at a high vacuum, and residual gas including the steam near the object formed membranes is lessened as much as possible, and film consistency lowering can be controlled.
[0011] Invention of claim 2 is characterized by being formed with at least one ceramic ingredient with which said 1st vacuum tub and a dielectric plate are chosen from boron nitride, boro-silicated glass, a quartz, oxidation aluminum, and nitriding aluminum in the formation equipment of an oxide optical thin film according to claim 1. The residual life of the radical within the 1st vacuum tub is prolonged because a radical makes said 1st vacuum tub and a dielectric plate the construction material by which a trap cannot be carried out easily, and a high-concentration radical can be supplied to the 2nd vacuum tub.
[0012] Invention of claim 3 is characterized by the oxygen radical concentration irradiated by the lifter in said 2nd vacuum tub formed membranes being more than 1016-piece [/cm] 2 and s in the formation equipment of claim 1 or an oxide optical thin film according to claim 2. mixing the evaporation particle from said evaporation source with a plasma style dominant in the high concentration oxygen radical 1016-piece [/cm] 2 and more than s -- high -- oxidation reaction can be promoted by the activity oxygen radical, and the surface impact by high energy particles, such as ion, can be controlled as much as possible, and the oxide thin film which has a high optical property can be produced.
[0013] Invention of claim 4 is the formation approach of the oxide optical thin film characterized by making into an evaporation source the evaporation material which becomes either of claim 1 to claims 3 from a metallic oxide using the formation equipment of the oxide optical thin film of a publication, and forming membranes to the lifter which does not carry out forcible heating formed membranes.
[0014] The object used by this invention formed membranes can be used irrespective of the construction material of a glass polymer film etc., or its configuration, and the effectiveness of this invention is not spoiled by the class of object formed membranes.
[0015]
[Embodiment of the Invention] The gestalt of operation of this invention is explained to a detail using a drawing below. Drawing 1 is approximate account drawing for explaining 1 operation gestalt of the formation equipment of the oxide optical thin film of this invention. Formation of the high refractive-index oxide optical thin film in this invention is performed using the vacuum evaporator 1 as shown in drawing 1 . This vacuum evaporator 1 The crucible 4 and evaporation material 3 which hold an evaporation material 3 in the vacuum chamber 2 are irradiated. The electron gun 5 to evaporate, The stainless steel tubing 6 inserted in the interior from the side attachment wall of the vacuum chamber 2, The ceramic tubing 8 around which the RF impression coil (an RF coil is called below) 7 installed in the interior of the stainless steel tubing 6 was wound, the dielectric ceramic

plate 9 with an orifice 14 installed in the point of the ceramic tubing 8, the membrane formation substrate (object formed membranes) 10, and it It consists of substrate electrode holders 11 to hold.
[0016] In case an oxide optical thin film is formed using the above-mentioned vacuum evaporator 1, the evaporation material 3 which consists of a metallic oxide held to the crucible 4 interior currently first installed in the lower part in the degree of vacuum 4x10 vacuum chamber 2 (the 2nd vacuum tub) set to -5 or less Pa is made into an evaporation source, and the evaporation material 3 is evaporated with the electron beam irradiated from an electron gun 5. On the other hand, oxygen gas is introduced into the ceramic tubing 8 (the 1st vacuum tub), and a pressure is set to about 30Pa. By carrying out the seal of approval of the RF output from RF coil 7, the oxygen plasma is generated and the oxygen plasma style 13 which has an atmosphere dominant in an oxygen radical through the orifice 14 of the dielectric ceramic plate 9 which exists at the head of the ceramic tubing 8 is irradiated toward the membrane formation substrate (object formed membranes) 10. At this time, the degree of vacuum in the vacuum tub 2 with the membrane formation substrate (object formed membranes) 10 is 10-3 to ten to 4 Pa, and is remarkably high from the degree of vacuum in the ceramic tubing 8. The evaporation particle 12 which evaporated from the evaporation source passes and mixes this oxygen plasma style 13, and a thin film is formed on the membrane formation substrate (object formed membranes) 10 held at the substrate electrode holder 11.
[0017]
[Example] Next, the example of this invention explains still more concretely. The glass plate was installed in the substrate electrode holder 11 of the upper part in the vacuum chamber 2 of a vacuum evaporation system 1 as a membrane formation substrate (object formed membranes) 10 first shown in drawing 1 . Subsequently, the evaporation material 3 was installed in the crucible 4. Membranes were formed on the following membrane formation conditions using the above-mentioned membrane formation substrate (object formed membranes) 10 and an above-mentioned evaporation source.
(1) electron beam output: -- 200 - 350W(2) RF-coil impression output: -- 450W(3) evaporation-material 3:TiOx (x=0.3-2.0)
(4) vapor rate: -- 0.5A[/s](5) system-of-reaction internal pressure: -- in addition, 8x10 - 4 or less Pa (object formed membranes) of membrane formation substrates 10 did not carry out forcible heating.
[0018] The refractive index and absorption coefficient of a thin film which formed membranes in the above-mentioned example were computed using the formula (J. Phys.E, 16, 1214 (1983) reference) of Swanepoel, after measuring a reflection factor and permeability. The refractive index, the absorption coefficient (%), and (the wavelength of 550nm) of a titanium oxide thin film which were created from each evaporation material are shown in a table 1.
[0019]
[A table 1]

蒸着材料		屈折率	吸収率（%）
TiO_x	0．3<x≦1．0	2．4	0．50
TiO_x	1．0<x≦1．5	2．3	0．44
TiO_x	1．5<x≦1．7	2．3	0．52
TiO_x	1．7<x≦2．0	2．3	0．49

[0020] All the titanium oxide thin films obtained from each evaporation material were transparence. The titanium oxide thin film which formed membranes as shown in a table 1 showed the high refractive index of 2.3 more than, and 0.52% or less of few absorption coefficients in the wavelength of 550nm, and had the outstanding optical property.
[0021] Moreover, about the titanium oxide thin film obtained from each evaporation material, the moisture-proof heat instability test under the situation of 60 degrees C of ambient temperature and 90% of humidity was performed. This carries out 1000 timing measurement of the peak wavelength change of the reflection factor of the thin film under the above-mentioned conditions. A refractive index changes because moisture sticks to the

hole in a titanium oxide thin film. As for change of a refractive index, the peak wavelength of the reflection factor it indicates optical thickness to be since optical thickness is proportional to a refractive index is shifted. That is, this trial serves as a rule of thumb of the precision of a titanium oxide thin film. As a result of measuring the above-mentioned peak wavelength change, it turned out that it is 5nm or less in all titanium oxide thin films, and is the very precise film.

[0022]

[Effect of the Invention] Although the high refractive-index oxide thin film was conventionally obtained only at hot substrate temperature (whenever [formed membranes temperature]), if the formation equipment of the oxide optical thin film of this invention is used, the membrane formation of an oxide optical thin film which has an optical property superior to the thin film obtained with the vacuum deposition method or plasma assistant vacuum deposition used from the former, without carrying out forcible heating of the object formed membranes will be attained. For this reason, the membrane formation of the high performance oxide optical thin film to the objects made difficult formed membranes, such as a plastic part and precision components, of an activity is attained conventionally, and industrial value is great.

[0023] The oxide optical thin film which has the optical property which was excellent by the formation approach of the oxide optical thin film of this invention, without carrying out forcible heating of the object formed membranes can be formed easily.

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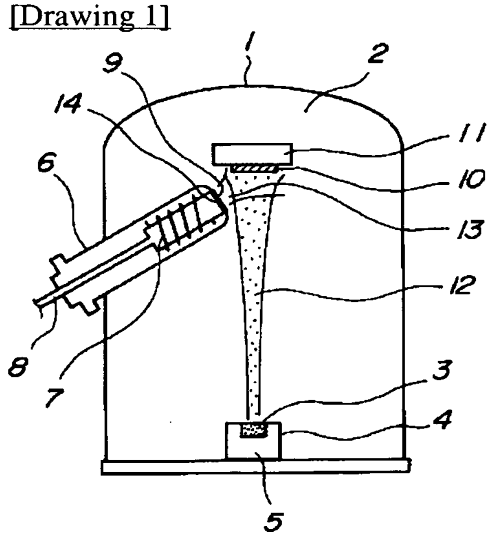
* NOTICES *

DRAWINGS

[Drawing 1]



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